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Published in:
 Inorganica Chimica Acta

DOI:
 [10.1016/j.ica.2016.10.021](https://doi.org/10.1016/j.ica.2016.10.021)

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Document Version
 Publisher's PDF, also known as Version of record

Publication date:
 2017

[Link to publication in University of Groningen/UMCG research database](#)

Citation for published version (APA):

Shaabani, B., Rad-Yousefnia, N., Zahedi, M., Ertan, Ş., Blake, G. R., & Zakerhamidi, M. S. (2017). Construction of new 1D and 2D coordination polymers generated from rigid N,N'-bis(4-pyridylmethylene)-1,5-naphthalenediamine ligand: Syntheses, crystal structures and luminescence properties. *Inorganica Chimica Acta*, 455(1), 158-165. <https://doi.org/10.1016/j.ica.2016.10.021>

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Research paper

Construction of new 1D and 2D coordination polymers generated from rigid N,N'-bis(4-pyridylmethylene)-1,5-naphthalenediamine ligand: Syntheses, crystal structures and luminescence properties

Behrouz Shaabani^{a,*}, Negar Rad-Yousefnia^a, Mansoureh Zahedi^a, Şahin Ertan^b, Graeme R. Blake^c, Mohammad Sadegh Zakerhamidi^d

^a Department of Inorganic Chemistry, Faculty of Chemistry, Tabriz University, Tabriz, Iran

^b Department of Chemistry, Faculty of Science, Ataturk University, Erzurum 25240, Turkey

^c Zernike Institute for Advanced Materials, University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands

^d Research Institute for Applied Physics and Astronomy, University of Tabriz, Tabriz, Iran

ARTICLE INFO

Article history:

Received 21 June 2016

Received in revised form 12 October 2016

Accepted 14 October 2016

Available online 15 October 2016

Keywords:

Coordination polymer

Dipyridyl ligand

Single crystal X-ray diffraction

Thermal behavior

Photoluminescence property

ABSTRACT

Treatment of N,N'-bis(4-pyridylmethylene)-1,5-naphthalenediamine (L) with $\text{Pb}(\text{OAc})_2/\text{KBr}$, $\text{Cu}(\text{acac})_2$, and $\text{Cu}(\text{OAc})_2$ afforded three new coordination polymers $[\text{Pb}(\mu\text{-L})(\mu\text{-Br})_2]_n$ (**1**), $[\text{Cu}(\mu\text{-L})(\text{acac})_2]_n$ (**2**), and $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**). These coordination polymers have been structurally characterized by single crystal X-ray diffraction. Compound **1** has a 2D sheet structure in which the lead(II) centers are bridged by both L and bromide ligands. Compound **2** adopts a 1D neutral coordination chain and consists of bis (2,4-pentanedionato) copper(II) units connected by rigid bridging L ligands. The structure of compound **3** also adopts a 1D neutral coordination chain in which two copper centers are connected through four acetate groups to form a $\text{Cu}(\text{OAc})_4\text{Cu}$ paddle-wheel-type cage between two bridging L ligands. The FT-IR spectra, thermal behavior and photoluminescence properties of these coordination polymers have also been investigated.

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1. Introduction

The rational design and construction of novel coordination polymers have attracted great academic and commercial interest in the field of supramolecular chemistry and crystal engineering in the last few decades. This is due to their intriguing variety of architectures and topologies, and promising applications in the fields of ion exchange [1], nonlinear optics [2], gas storage [3], luminescence [4], conductivity [5], heterogeneous catalysis [6] and magnetism [7]. One of the ultimate goals of crystal engineering is to synthesize coordination polymers with interesting structures and desired properties. The structures and observed properties of coordination polymers can be affected by different factors such as multidentate ligands, the type of metal ions, solvent effects, and the ratio of metal-to-ligand [8–13].

Among these parameters, the organic linkers also play a key role in determining the diverse architectures of coordination polymers due to their length, rigidity or flexibility, and the number, type and angle of donor atoms [14,15]. So far, a large number of coordina-

tion polymers have been reported based on 4,4'-bipyridyl-type ligands. Neutral 4,4'-bipyridyl-based ligands with different spacers between the bipyridine functionalities are extremely versatile and proficient ligands that can construct diverse coordination polymers with interesting structures such as one-dimensional chains and ladders, two-dimensional layers and three-dimensional frameworks [16–20].

In this research, we have used a rigid, long π -conjugated analogue of the 4,4'-bipyridine linker, N,N'-bis(4-pyridylmethylene)-1,5-naphthalenediamine (Scheme 1). To the best of our knowledge, including a search in the Cambridge Structural Database (CSD), only fourteen metal complexes with this ligand have been reported [21–27]. Lee's group have prepared three diamondoid 3D framework from silver salts (AgX : $\text{X} = \text{PF}_6$, SbF_6 , BF_4) and rigid L ligand [22]. Also, this group have utilized this ligand to prepare zero- to three-dimensional compounds: dinuclear platinum compound, $[\text{Pt}_2\text{L}(\text{SMe}_2)_2\text{Cl}_4]$ [23], 1-D zigzag-chain coordination polymer $[\text{Zn}(\text{L})(\text{NO}_3)_2]$, two 1-D ladder structures $[\text{Cd}(\text{L})_{1.5}(\text{NO}_3)_2]$, and $[\text{CoL}_{1.5}(\text{NO}_3)_2]$, 2D mixed-ligand coordination polymer $[\text{Cd}_2(\text{L})_2(\text{L})(\text{NO}_3)_4]$, and a 3-D compound $[\text{CoL}(\text{bpdc})](\text{EtOH})$ [21,24,27]. Three 1D and 2D iron coordination polymers were recently reported by Kepert and co-workers [25]. So far, any compound

* Corresponding author.

E-mail address: shaabani.b@gmail.com (B. Shaabani).

Scheme 1. Schematic illustration of ligand used in this study.

containing lead or copper cation is not reported with this ligand. These compounds may be having different structures and interesting properties due to role of the metal ion comparable to above-mentioned coordination polymers. We report here on the syntheses, crystal structures, and thermal and luminescent properties of three new coordination polymers assembled using the *N,N'*-bis(4-pyridylmethylene)-1,5-naphthalenediamine ligand and some metal salts ($\text{Pb}(\text{OAc})_2/\text{KBr}$, $\text{Cu}(\text{acac})_2$, and $\text{Cu}(\text{OAc})_2$), namely $[\text{Pb}(\mu\text{-L})(\mu\text{-Br})_2]_n$ (**1**), $[\text{Cu}(\mu\text{-L})(\text{acac})_2]_n$ (**2**), and $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**).

2. Experimental

2.1. Materials and physical measurements

$\text{Cu}(\text{acac})_2$ was prepared according to a literature method [28]. All other reagents and solvents were obtained from commercial sources and used as received. Elemental analyses were obtained on an Elementar Vario EL III. Fourier Transform Infrared (FT-IR) spectra were recorded in KBr pellets using a Bruker Tensor 27 FT-IR spectrometer. The thermal behaviors of all coordination polymers were measured with a Linseis- L81-A 1750 apparatus. Photoluminescence spectra were taken with JASCO FP-6200 luminescence spectrometer.

2.2. Syntheses of compounds

2.2.1. Synthesis of the ligand *N,N'*-bis(4-pyridylmethylene)-1,5-naphthalenediamine (**L**)

An ethanol (10 ml) solution of 4-pyridine carboxaldehyde (1.88 ml, 20.0 mmol) was added dropwise to an ethanol solution (10 ml) of 1,5-diaminonaphthalene (1.58 g, 10.0 mmol) and the mixture was refluxed for 2 h. The resulting yellow crystalline solid was separated and filtered off, then washed with ethanol (3×5 ml) and hexane (3×5 ml), to give ligand **L**. Yield: 83% (2.80 g). M.p.: $>200^\circ\text{C}$. FT-IR (KBr, cm^{-1}): 3028(w), 1621 (s), 1590 (s), 1403 (s), 1313 (m), 1225 (s), 975 (m), 921 (m), 786 (s), 647 (w). *Anal.* Calc. for $\text{C}_{22}\text{H}_{16}\text{N}_4$: C, 78.6; H, 4.8; N, 16.7. Found: C, 78.4; H, 4.6; N, 16.5%.

2.2.2. Preparation of $[\text{Pb}(\mu\text{-L})(\mu\text{-Br})_2]_n$ (**1**)

For obtaining single crystals of compound **1**, we used a thermal gradient method in a branched tube [29,30]. The ligand **L** (0.2 mmol, 0.067 g), lead(II) acetate (0.2 mmol, 0.065 g) and potassium bromide (0.4 mmol, 0.047 g) were placed at the bottom of the main arm of a glass tube which then was slowly filled with methanol. The tube was sealed and the arm containing the reactants was immersed in an oil bath at 60°C while the other arm was kept at ambient temperature. Yellow crystals of **1** were obtained after one day and were filtered off and dried in air. Yield: 17.7% (0.025 g) relative to Pb. FT-IR (KBr, cm^{-1}) bands: 3040(w), 3024 (w), 1601(s), 1552(m), 1542(m), 1418(m), 1403(m), 1363(m), 1203(m), 1057(m), 1000(s), 974 (m), 822(s), 776(vs). *Anal.* Calc.

for $\text{C}_{22}\text{H}_{16}\text{N}_4\text{Br}_2\text{Pb}$: C, 37.6; H, 2.3; N, 8.0. Found: C, 37.7; H, 2.5; N, 7.8%.

2.2.3. Preparation of $[\text{Cu}(\mu\text{-L})(\text{acac})_2]_n$ (**2**)

A 2.5 ml chloroform solution of **L** (0.25 mmol, 0.084 g) was added drop wise to an 8 ml ethanol-chloroform solution (1:1, v/v) of $[\text{Cu}(\text{acac})_2]$ (0.25 mmol, 0.065 g) and the resulting solution was stirred for one hour. The color of the solution immediately changed from blue to bright green on adding the **L** ligand. The resulting clear solution was kept at room temperature while the solvent evaporated slowly. Green crystals of **2** were obtained after 8 days. Yield: 71.8%, (0.107 g) relative to Cu. FT-IR (KBr, cm^{-1}) bands: 3043(w), 2922(w), 2854(w), 1581(s), 1520(s), 1457 (m), 1404(s), 1326(m), 1263(w), 1228(w), 992(w), 926(w), 785 (m). *Anal.* Calc. for $\text{C}_{32}\text{H}_{30}\text{N}_4\text{O}_4\text{Cu}$: C, 64.3; H, 5.1; N, 9.4. Found: C, 64.5; H, 5.3; N, 9.5%.

2.2.4. Preparation of $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**)

A mixture of dichloromethane and methanol (1:1, 2 ml) was gently layered on the top of a dichloromethane solution (8 ml) of **L** (0.034 g, 0.1 mmol) in a test tube. A solution of $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (0.02 g, 0.1 mmol) in MeOH (8 ml) was added carefully as a third layer. Green crystals were obtained after four days. Yield: 18.60%, (0.013 g) relative to Cu. FT-IR (KBr, cm^{-1}) bands: 3055(w), 3009 (w), 2978(w), 2927(w), 2873(w), 1618(vs), 1608(sh), 1580(w), 1558(w), 1507(w), 1432(vs), 1413(sh), 1318(w), 1235(w), 830 (w), 797(w), 682(m). *Anal.* Calc. for $\text{C}_{30}\text{H}_{28}\text{N}_4\text{O}_8\text{Cu}_2$: C, 51.5; H, 4.0; N, 8.0. Found: C, 51.2; H, 4.1; N, 8.3%.

2.3. X-ray crystallography

For compound **1**, $[\text{Pb}(\mu\text{-L})(\mu\text{-Br})_2]_n$, yellow columnar crystals were analyzed using a Bruker D8 Venture diffractometer operating with $\text{MoK}\alpha$ radiation and equipped with a Photon100 area detector and a Triumph monochromator. Data were collected at 100(2) K. The data were processed using the Bruker Apex II software, the structure was solved by direct methods, and the resulting structural parameters were refined using the SHELXTL software. Hydrogen atoms were generated by geometrical considerations and constrained to their idealized positions. The X-ray diffraction data for the crystals of compounds **2** and **3** were collected on a four-circle Rigaku R-Axis RAPID-S diffractometer equipped with a two-dimensional area IP detector with graphite monochromated $\text{MoK}\alpha$ ($\lambda = 0.71073 \text{ \AA}$) radiation at room temperature. The lattice parameters were determined by the least-squares methods on the basis of all reflections with $F^2 > 2\sigma(F^2)$. Integration of the intensities, correction for Lorentz and polarization effects and cell refinement was performed using CrystalClear (Rigaku/MSC Inc., 2005) software [31]. The structures were solved by direct methods using SHELXS-97 [32] and refined by a full-matrix least-squares procedure using the program SHELXL-97 [31]. All non-hydrogen atoms were refined anisotropically and H atoms were positioned geometrically and refined using a riding model. The final difference Fourier maps showed no peaks of chemical significance. A summary of crystallographic data, experimental details, and refinement results for compounds is listed in Table 1. Selected bond lengths and angles are given in Table 2. Crystallographic data that were deposited in CSD under CCDC-1481783 (for **1**), 1481155 (for **2**) and 1481350 (for **3**) registration numbers contain the supplementary crystallographic data for this Letter. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre (CCDC) via www.ccdc.cam.ac.uk/data_request/cif and are available free of charge upon request to CCDC, 12 Union Road, Cambridge, UK (fax: +44 1223 336033, e-mail: deposit@ccdc.cam.ac.uk).

Table 1
X-ray data collection and structure refinement details.

	1	2	3
Formula	C ₁₁ H ₈ BrN ₂ Pb _{0.50}	C ₃₂ H ₃₀ CuN ₄ O ₄	C ₁₅ H ₁₄ CuN ₂ O ₄
fw	351.70	598.14	349.82
Temperature (K)	100(2)	293(2)	293(2)
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	<i>P2</i> / <i>c</i>	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
<i>a</i> (Å)	16.0660(11)	8.639(2)	7.4697(8)
<i>b</i> (Å)	4.1425(3)	8.872(2)	8.0450(8)
<i>c</i> (Å)	17.5091(12)	10.895(4)	13.0198(13)
α°	90	79.630(5)	96.433(4)
β°	112.764(2)	73.048(4)	98.105(4)
γ°	90	65.500(5)	93.108(4)
<i>V</i> (Å ³)	1074.52(13)	725.2(4)	767.76(14)
<i>Z</i>	4	1	2
<i>D</i> _{cal} (g cm ^{−3})	2.174	1.370	1.513
μ (mm ^{−1})	11.59	0.80	1.44
<i>F</i> (0 0 0)	656	311	358
<i>T</i> _{min}	0.0357	0.657	0.657
<i>T</i> _{max}	0.0788	0.746	0.746
2 θ range (°)	2.926–24.713	2.99–28.35	2.976–28.356
Number of reflections measured	21,604	35,373	31,548
Number of unique reflections	1823	3603	3824
Number of reflections with <i>I</i> > 2 σ (<i>I</i>)	1660	3297	3260
Number of parameters refined	132	189	201
Maximum in $\Delta\rho$ (e Å ^{−3})	1.60	0.276	0.45
Minimum in $\Delta\rho$ (e Å ^{−3})	−2.93	−0.374	−0.32
Goodness-of-fit on <i>F</i> ²	1.115	1.123	1.046
<i>R</i> ₁	0.0276	0.0307	0.0312
w <i>R</i> ₂	0.0569	0.0853	0.0825
<i>R</i> _{int}	0.048	0.027	0.038

3. Results and discussion

3.1. Syntheses of the compounds and FT-IR spectra

The relatively long, rigid 4,4'-bipyridine-type bridging ligand was prepared by the condensation reaction of 1,5-diaminonaphthalene and 4-pyridinecarboxyaldehyde in high yields and purity. Yellow single crystals of coordination polymer [Pb(μ -L)(μ -Br)₂]_n (**1**) were obtained from the reaction among ligand L, lead(II) acetate and potassium bromide in a molar ratio of 1:1:2 by a branched tube method. Also, bright green single crystals of [Cu(μ -L)(acac)₂]_n (**2**) were obtained by slow evaporation from the reaction of Cu(acac)₂ dissolved in ethanol-chloroform solution with bridging L ligand in chloroform in a 1:1 M ratio. Finally the reaction of Cu(OAc)₂·H₂O with ligand L in CH₃OH/CH₂Cl₂ in a 1:1 M ratio resulted in the formation of a new dicopper 1D neutral chain [Cu₂(μ -L)(μ -OAc)₄]_n (**3**). All obtained coordination polymers have been characterized by photoluminescence and FT-IR spectroscopic methods, TGA and DTA, and single crystal X-ray diffraction.

FT-IR spectra of these compounds display the characteristic relatively weak absorption bands around 2854–3055 cm^{−1} due to the C–H modes. The FT-IR spectra of coordination polymers **1**, **2**, and **3** display C=N stretching frequencies at 1601, 1581, and 1608 cm^{−1} respectively, consistent with the fact that $\nu_{C=N}$ in the free Schiff base appears at 1621 cm^{−1} that is at the common frequency range for imine bands [33–35]. The azomethine (C=N) stretching frequency of **2** overlaps with $\nu_{C=O}$ in acac. The FT-IR spectrum of **2** shows two carbonyl stretching bands at 1581 and 1520 cm^{−1}; these stretching bands in Cu(acac)₂ appear at 1577 and 1529 cm^{−1}, respectively [36]. For **3**, two sharp and strong characteristic bands at 1618 [ν_{as} (COO)] and 1432 cm^{−1} [ν_{sym} (COO)]

Table 2
Selected bond lengths Å and angles (°).

Compound 1			
Pb1–N1A	2.663(5)	Pb1–Br2 ⁱ	2.9524(7)
Pb1–N1A ⁱ	2.663(5)	Pb1–Br2 ⁱⁱ	3.0159(7)
Pb1–Br2	2.9524(7)	Pb1–Br2 ⁱⁱⁱ	3.0159(7)
N1A–Pb1–N1A ⁱ	179.6(3)	Br2–Pb1–Br2 ⁱⁱ	178.74(2)
N1A–Pb1–Br2	87.87(12)	Br2 ⁱ –Pb1–Br2 ⁱⁱ	87.903(16)
N1A ⁱ –Pb1–Br2	91.88(12)	N1A–Pb1–Br2 ⁱⁱⁱ	88.16(12)
N1A–Pb1–Br2 ⁱ	91.88(12)	N1A ⁱ –Pb1–Br2 ⁱⁱⁱ	92.09(12)
N1A ⁱ –Pb1–Br2 ⁱ	87.87(12)	Br2–Pb1–Br2 ⁱⁱⁱ	87.903(16)
Br2–Pb1–Br2 ⁱ	93.36(3)	Br2 ⁱ –Pb1–Br2 ⁱⁱⁱ	178.74(2)
N1A–Pb1–Br2 ⁱⁱ	92.09(12)	Br2 ⁱⁱ –Pb1–Br2 ⁱⁱⁱ	90.83(3)
N1A ⁱ –Pb1–Br2 ⁱⁱ	88.16(12)	–	–
Symmetry codes: (i) $-x, y, -z + 1/2$; (ii) $-x, y-1, -z + 1/2$; (iii) $x, y-1, z$; (iv) $x, -y + 1, z-1/2$; (v) $x, -y + 1, z + 1/2$; (vi) $x, y + 1, z$; (vii) $-x + 1, -y + 2, -z + 1$			
Compound 2			
Cu–O2 ⁱ	1.950(2)	Cu–O1 ⁱ	1.951(2)
Cu–O2	1.950(2)	Cu–N1	2.484(2)
Cu–O1	1.952(2)	Cu–N1 ⁱ	2.484(2)
O2–Cu–O2 ⁱ	180.0	O1–Cu–N1	93.16(5)
O2–Cu–O1 ⁱ	86.86(5)	O2–Cu–N1	87.70(5)
O2–Cu–O1	93.14(5)	O1–Cu–N1 ⁱ	86.84(5)
O2 ⁱ –Cu–O1 ⁱ	93.14(6)	O2–Cu–N1 ⁱ	92.30(5)
O2–Cu–O1 ⁱ	86.86(6)	N1–Cu–N1 ⁱ	180.0
O1–Cu–O1 ⁱ	180.0	O1 ⁱ –Cu–N1 ⁱ	93.16(5)
O1 ⁱ –Cu–N1	86.84(5)	O2 ⁱ –Cu–N1 ⁱ	87.70(5)
Symmetry codes: (i) 1– <i>x</i> , 1– <i>y</i> , 2– <i>z</i>			
Compound 3			
Cu–O4 ⁱ	1.9644(15)	Cu–O3	1.9710(15)
Cu–O1	1.9701(15)	Cu–N1	2.1877(16)
Cu–O2 ⁱ	1.9699(16)	Cu–Cu ⁱ	2.6208(5)
O4 ⁱ –Cu–O1	89.44(7)	O4 ⁱ –Cu–O3	168.48(6)
O4 ⁱ –Cu–O2 ⁱ	89.27(7)	O1–Cu–O3	90.48(7)
O1–Cu–O2 ⁱ	168.57(6)	O2 ⁱ –Cu–O3	88.54(7)
O4 ⁱ –Cu–N1	98.87(6)	O1–Cu–N1	93.57(6)
O2 ⁱ –Cu–N1	97.85(7)	O3–Cu–N1	92.63(6)
Symmetry codes: (i) $-x + 2, -y + 1, -z + 1$			

can be assigned to the acetate group vibrations. The Δ value ($\nu_{as}(\text{COO}) - \nu_{sym}(\text{COO})$) indicates that the acetate anion coordinates to the Cu(II) centers in bridging mode [36,37].

3.2. Description of crystal structures

3.2.1. [Pb(μ -L)(μ -Br)₂]_n (**1**)

X-ray diffraction analysis of yellow columnar crystals of **1** at 100 K shows that this compound crystallizes in the monoclinic system with space group *P2*/*c*. There is one crystallographically independent lead(II) ion that adopts a distorted octahedral geometry with a N₂Br₄ environment (Fig. 1) from four bromide anions and two L ligands. The twofold rotation axis along the crystallographic *b*-axis passes through lead ion between the two bromide atoms, and the glide plane is perpendicular to it. The inversion center is also at the center of the naphthalene ring of L Ligand. Adjacent Pb atoms are linked through pairs of bromide anions, yielding distorted PbBr₂ rhombi, to form 1D chains along the *b*-axis (Fig. 2). The adjacent Pb···Pb distance in the PbBr₂ building block is 4.1425(3) Å, which defines the *b*-axis. Despite the presence of only one crystallographically independent Br atom, the lead-bromide distances alternate between 2.9524(7) Å and 3.0159(7) Å along the *b*-axis and the Br–Pb–Br bond angles vary from 87.902(16)° to 178.74(2)°. Extensive cross linking occurs via bidentate bridging L ligands which lie along the *ac* plane to produce a 2D framework (Fig. 3) with bond distances Pb(1)–N(1A) and Pb(1)–N(1Aⁱ) = 2.663(5) Å. There are neither any intermolecular $\pi \cdots \pi$ interactions between rings of the L ligands nor hydrogen bonds, and adjacent 2D-layers are held together only by Van der Waals interactions. The angular distribution of ligands around the lead cation is symmetrical and there are no identifiable voids around the coordination sphere of

Fig. 1. Coordination environment of lead(II) in **1**.

Fig. 2. The PbBr_2 building block of **1**, forming 1D chains along the *b*-axis.

lead, implying no stereochemically active lone pair. Therefore, the six-coordinate lead atom has a holodirected coordination geometry. Pb(II) complexes with intermediate coordination numbers (6–8) can show either holodirected or hemidirected coordination geometries. A holodirected geometry is preferred when the ligands are soft and bulky or have strong interligand repulsion [38]. The holodirected structure in coordination polymer **1** is probably due to the existence of four relatively soft and negatively charged bromide ligands that repel each other and two very bulky L ligands. Analogous structures to **1** were previously reported for $[\text{Pb}(\mu\text{-4-bpdb})(\mu\text{-Br})_2]_n$ (4-bpdb is 1,4-bis(4-pyridyl)-2,3-diaza-1,3-butadiene) [12], $[\text{Pb}(\mu\text{-bpp})(\mu\text{-Br})_2]_n$ (bpp is 1,3-di(4-pyridyl)propane) [39], and $[\text{Pb}(\mu\text{-bdabpm})(\mu\text{-Br})_2]_n$ (bdabpm is 1,4-benzenediamine, *N,N'*-bis(3-pyridinylmethylene)) [40].

3.2.2. $[\text{Cu}(\mu\text{-L})(\text{acac})_2]_n$ (**2**)

Compound **2** crystallizes in the triclinic system with space group $P\bar{1}$. The molecular unit of **2** is shown in Fig. 4. It is a 1D neutral coordination chain and consists of bis(2,4-pentanedionato)-copper(II) units connected by rigid bridging L ligands. The coordination geometry of the Cu(II) center can be described as a distorted octahedron with Jahn-Teller distortion. The copper atom is located on an inversion center and is coordinated to four oxygen atoms from two acac ligands and two nitrogen atoms from two different L ligands. Also, the other inversion center is located at the center of the naphthalene ring of L Ligand. Four oxygen atoms occupy the equatorial plane with bond lengths of 1.950(2) and 1.95(2) Å, while two nitrogen atoms occupy the axial positions at 2.484(2) Å. The O1–Cu–O2, O1–Cu–N1, O2–Cu–N1 bond angles deviate only slightly from the ideal value of 90°, and the O1–Cu–O1, O2–Cu–O2, N1–Cu–N1 bond angles are equal to the ideal value of 180°. The bidentate bridging L ligand is connected to two $[\text{Cu}(\text{acac})_2]$ units to form a 1D chain with the nearest Cu···Cu separation 20.623(2) Å within the chain. Each chain is surrounded by two other chains (Fig. 5). In the solid state (Fig. S1), the chains are interconnected through weak hydrogen-bonding interactions involving the O(1) from acac ligands with the H(9), and H(11) atoms on the L ligands of the neighboring molecule. The O(1)···H(9), and O(1)···H(11) distances are 2.670, and 2.686 Å, respectively. The coordination polymeric chains are also connected via intermolecular $\pi\cdots\pi$ interactions between C9 and C10 of the pyridine rings of two adjacent chains at a distance of 3.303 Å to generate a 2D sheet and this distance is shorter than a distance of 3.396(6) Å in $[\text{Cu}(\text{tfacac})_2(4,4'\text{-bpe})]$ [41].

3.2.3. $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**)

In the 1D neutral chain $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**), two copper centers are connected through four acetate groups to form a $\text{Cu}(\text{OAc})_4\text{Cu}$ paddle-wheel-type cage between two bridging L

Fig. 3. A fragment of the two-dimensional coordination polymer in **1**.

Fig. 4. A fragment of the 1D coordination polymer **2**. Thermal ellipsoids are drawn at the 30% probability level.

Fig. 5. 2D network packing diagram of coordination polymer **2**.

ligands. This compound also crystallizes in the triclinic system with space group $P\bar{1}$. As shown in Fig. 6, the copper center is penta coordinated and adopts a square pyramidal geometry with a CuO_4N chromophore. The four coordination sites of Cu(II) in the equatorial plane are occupied by four oxygen atoms from four distinct acetate anions with the Cu-O bonds in the range of 1.9644(15)–1.9710 (15) Å. The $\text{O-Cu-O}_{\text{cis}}$ bond angles vary from $88.54(7)^\circ$ to $90.48 (7)^\circ$ and the $\text{O-Cu-O}_{\text{trans}}$ angles are $168.48(6)^\circ$ and $168.57(6)^\circ$. The apical position is occupied by one nitrogen atom from the pyridyl ring of **L** with a Cu-N distance of 2.1877(16) Å and O-Cu-N angles in the range of $92.63(6)^\circ$ – $98.87(6)^\circ$. The two copper centers within the $[\text{Cu}_2(\mu\text{-OAc})_4]$ binuclear building unit are related by an inversion center with a very short adjacent $\text{Cu}\cdots\text{Cu}$ distance (2.6208 (5) Å) which is in the common range of reported complexes containing similar paddle-wheel-type cage [42,43]. The nature of the $\text{Cu}\cdots\text{Cu}$ interaction has been the subject of controversy. For

example, Figgis and Martin have proposed a direct metal-metal δ -bond between the two adjacent copper cations, which is formed by lateral overlap of $3d_{x^2-y^2}$ orbitals [44]. Forster and Ballhausen have suggested the existence of a rather strong σ -band between them [45]. On the other hand, structural and magnetic studies on a pair of analogous copper(II) acetate and formate dimers demonstrate the insensitivity of the magnetic exchange coupling constant (J) to the $\text{Cu}\cdots\text{Cu}$ distance. It seems that the carboxylate bridges play a key role in the transfer of antiferromagnetic coupling between the paramagnetic centers [46]. Therefore the direct copper-copper interaction in analogous dimeric structures can be assumed very weak or even negligible. Also, the bridging **L** ligand is located on an inversion center. Stability of crystal lattice in **3** is completed via intermolecular $\text{C(9)-H(9)}\cdots\text{O(3)}$ hydrogen bonds (the $\text{O(3)}\cdots\text{H(9)}$ bond length is 2.602 Å) in which the oxygen atoms of acetate anion act as hydrogen bond acceptors. There are very weak $\pi\cdots\pi$

Fig. 6. View of 1D chain structure in compound **3**.

interactions between C(2)···C(11) and C(2)–H(2)···C(11) at a distance of 3.397 Å, and 2.792 Å, respectively (Fig. S2).

3.3. Thermal stabilities

To examine the thermal stability of coordination polymers **1–3**, thermo gravimetric and differential thermal analyses (TGA and DTA) were carried out in a nitrogen atmosphere. Compound **1** displays remarkable thermal stability up to 350 °C (Fig. S3). The TGA curve shows weight loss in the temperature range 350–403 °C, corresponding to two bromide anions (obsd. 22.13%, calcd. 22.70%), with an endothermic peak at 374 °C. Subsequent to this, a second weight loss occurs from 403 to 756 °C, which is due to sublimation of the product formed in the first step. A broad endothermic peak is observed in the DTA curve with a minimum at 640 °C. However, sublimation is still not complete at 756 °C, as indicated by the slope of the TGA curve. The TGA/DTA study of crystalline compound **2** indicates that the framework is stable up to 182 °C (Fig. S4). A first weight loss of 15.55% (calcd. 16.57%) occurs from 182 to 230 °C corresponding to the loss of one acac chelating ligand per formula unit with a sharp, endothermic DTA peak at 195 °C. In the range 230–405 °C the loss of the second acac anion is observed (obsd. 16.52%, calcd. 16.57%) with a much broader endothermic signal (the minima are at 242 and 375 °C). A third stage of mass loss of 51.27% takes place in the range 405–1000 °C corresponding to the L (calcd. 56.23%) ligand. The solid residue obtained at the maximum temperature of 1000 °C is suggested to be CuO (obsd. 15.94%, calcd. 13.30%). For compound **3**, no weight loss is observed up to 210 °C (Fig. S5). The TGA curve of **3** displays two distinct weight loss steps. The first step between 210 and 278 °C with a mass loss of 30.95% corresponds to the loss of 4 mol of the acetate anion (calcd. 33.70%). The second stage of mass loss (39.82%) takes place at 278–756 °C corresponding to a fragment (82.82%) of the L ligand. The solid residue obtained at the maximum temperature of 756 °C may correspond to 2 mol of CuO and the remaining (17.18%) L ligand (obsd. 29.44%, calcd: 30.99%). The DTA curve indicates that

the entire thermal decomposition of **3** takes place as an endothermic process.

3.4. Luminescence properties

The luminescence properties of **1–3** have been explored in the solid state at room temperature (Fig. 7). The free L ligand exhibits a broad emission band at 473 nm upon excitation at 350 nm, which can probably be attributed to $\pi \cdots \pi^*$ charge transfer. Coordination polymer **1** exhibits a broad, green luminescent emission band with a maximum at 511 nm ($\lambda_{\text{ex}} = 355$ nm) spreads from blue to green wavelengths (475–580 nm) and can be attributed to ligand-to-metal charge transfer (LMCT) between delocalized π bonds of the aromatic L ligands and the p orbitals of Pb(II) ions [47,48]. The novel lead(II) coordination polymer **1** may be a potential candidate for the emitting layer in organic light-emitting diodes (OLEDs) due to the broad luminescence emission peak, electron accumulation, conjugated structure, and stability under humid air and high temperature [49–54]. Compounds **2** and **3** show luminescent emission maxima at 476, 488 nm (**2**) and

Fig. 7. Normalized solid-state emission spectra of free L ligand, **1**, **2** and **3** at room temperature.

468 nm (**3**) when the excitation wavelengths are 360, and 380 nm, respectively. The emission bands at 466 and 464 nm are comparable to that of the corresponding ligand and can be ascribed to the intraligand charge transfer. Compound **2** also shows a bathochromic shift relative to the free L ligand and may be attributed to a ligand-to-metal charge transfer.

4. Conclusion

In summary, we have presented three coordination polymers $[\text{Pb}(\mu\text{-L})(\mu\text{-Br})_2]_n$ (**1**),

$[\text{Cu}(\mu\text{-L})(\text{acac})_2]_n$ (**2**) and $[\text{Cu}_2(\mu\text{-L})(\mu\text{-OAc})_4]_n$ (**3**) based on a rigid bidentate linker, N,N' -bis(4-pyridylmethylene)-1,5-naphthalenediamine. The structure of these coordination polymers has been determined by single-crystal X-ray diffraction and they have been characterized by IR, thermal gravimetric and differential thermal analyses, as well as luminescence measurements. Coordination polymer **1** has a 2D sheet structure while compounds **2** and **3** contain 1D neutral coordination chains. The lead coordination polymer exhibits higher thermal stability than the two copper coordination polymers. Compounds **2** and **3** have very similar thermal stability and luminescence spectra, while **1** exhibits a long-lived emission band at room temperature. This observation suggests that the metal ion plays a key role in the luminescent emission of our compounds. Further studies on rigid 4,4'-bipyridine-type bridging ligands are currently underway in our lab.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.jica.2016.10.021>.

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